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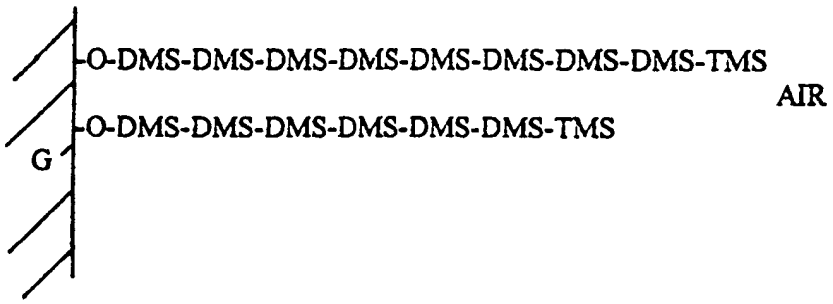

(57) Abstract

A silicone film is attached to a surface by chemical bonding. The silicone film consists of chains of siloxane groups, each chain terminating in an end molecule which is either an ester, an ether, or a halogen. The end molecule is allowed to react with water to produce an OH group. The surface is then contacted with a capping agent which reacts with the OH group to produce a new end group which improves the properties of the film.

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CAPPED SILICONE FILM AND METHOD OF MANUFACTURE THEREOF

Field of the Invention

This invention relates to the art of manufacturing silicone films.

More particularly, this invention relates to a method of manufacturing silicone films for coating surfaces containing OH or nitrogen hydrogen bonds.

In a further and more specific aspect, the instant invention concerns an improved method of manufacturing a water-repellent silicone film for coating glass and other surfaces.

Description of the Prior Art

Various methods exist for manufacturing easily cleanable, water repellent glass products, including shower doors, windshields, glass entry doors and glass partitions in restaurants. Two such methods are disclosed in U.S. Patents no 5,415,927 to Hirayama et el. and 4,263,350 to Valimont.

In another method currently in use, the glass is coated with a film consisting of chains of silicone molecules, with each chain chemically bound at one end to the surface of the glass. Each chain contains from dozens to hundreds of dimethylsiloxane (DMS) units and is terminated at its free end by either a hydroxyl (OH) group or a chlorine attached to a silicon, which soon reacts with water vapor in the air to produce OH groups. This existing film is in use on a number of glass products as well as other silica-containing products such as granite, porcelain, earthenware and stoneware, and for the most part, has performed satisfactorily. However, the water-repellence of the film is limited to some extent by the presence of

the terminal OH groups, which are highly water-attracting.

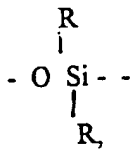
Accordingly, it is an object of the present invention to improve the water-resistance of silicone films on glass, and to provide a support film for chemically active substances.

Another object of the invention is to produce a family of silicone films for treating a variety of products such as the silica-containing products previously mentioned as well as organic substances including paper, cotton, nylon, leather, and wood, in order to improve the surface properties of those products.

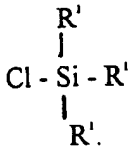
SUMMARY OF THE INVENTION

Briefly, to achieve the desired objects of the instant invention in accordance with the preferred embodiments thereof, a silicone film is attached to a surface by chemical bonding. The silicone film consists of chains of siloxane groups, each chain terminating in an end molecule which is either an ester, an ether, or a halogen. The end molecule is allowed to react with water in the surrounding air to produce an OH group. The surface is then contacted with a capping agent which reacts with the OH group to produce a new end group which improves the properties of the film.

The specific improvement in properties will depend on the siloxane groups used, as well as the composition of the capping agent. In general, the siloxane groups have the formula



and the capping agent has the formula



For water-repellent applications, R consists of nonpolar groups, and R' consists of inert groups. For non-water-repellent applications, R consists of polar groups. In other applications, R' could consist of chemically active groups, enabling the surface to be used as a solid state ion exchanger or an attachment point for chemically bound enzymes, chelating agents, dyes, chemical indicators or other substances.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and further and more specific objects and advantages of the instant invention will become readily apparent to those skilled in the art from the following detailed description of the preferred embodiments thereof taken in conjunction with the drawings in which:

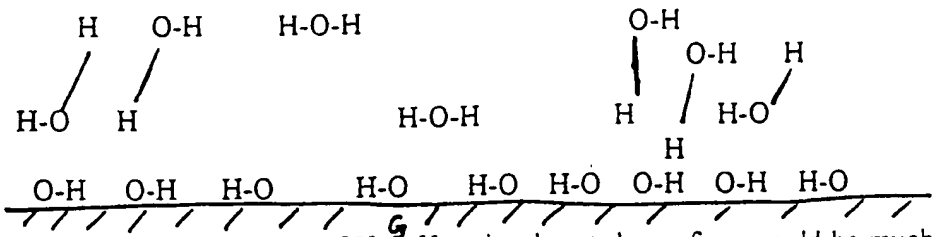
FIG. 1 is a diagrammatic representation of a surface coated with a prior art water-repellent film; and

FIG. 2 is a diagrammatic representation of a surface coated with a water-repellent film manufactured using the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Turning now to the drawings, attention is first directed to FIG. 1, which shows a surface G which has been treated with a water-repellent film using a prior art process. In the most widely-used application of the process, the surface G is glass, but the process may actually be used to treat any surface containing OH or nitrogen hydrogen bonds, such as silica-containing surfaces including granite, porcelain, earthenware and stoneware, as well as organic substances including cotton, paper, nylon, leather and others. The film comprises chains of dimethylsiloxane (DMS) groups. Each chain is chemically bonded at one end to an oxygen (O) molecule, which in turn is chemically bonded to the surface G. The opposite end of each chain includes either a hydroxyl (OH) group or a chlorine attached to silicone, which will soon react with water vapor in the surrounding air to produce an OH group.

The process by which the film of FIG. 1 is created is as follows. Initially, the surface G is moistened. The moistened surface can be represented as shown in simplified form below:

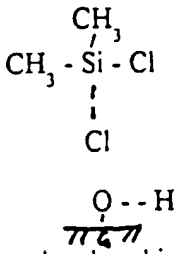


In reality, however, the number of H-O-H molecules at the surface would be much greater than the number of O-H groups on the surface (a ratio of about 100:1).

Next, the surface is treated with dimethyldichlorosilane using Portable Vapor machines which may be adapted to fixed site chambers for large volume operations, or by using a wipe-on

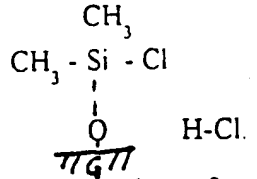
method or a dipping or spraying procedure. Where necessary, cyclohexylamine is used as a primer to ensure sufficient moisture for the chemical reaction to take place. After the dimethyldichlorosilane has been applied, a dimethyldichlorosilane molecule approaches an O-H group at the surface, as shown below:

(a)



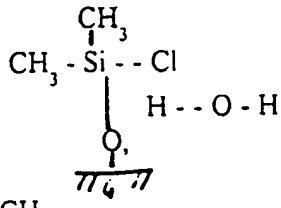
The ensuing reaction results in an anchor molecule which will chemically bond the film to the surface G, as shown below:

(b)



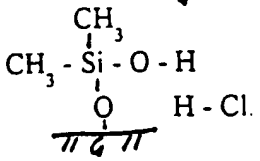
The Si - Cl bond then reacts with water absorbed on the surface G as follows:

(c)



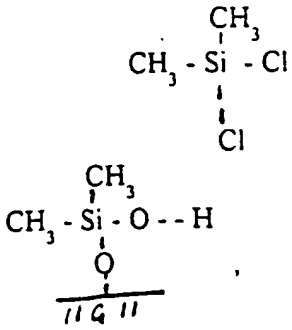
resulting in the following structure:

(d)

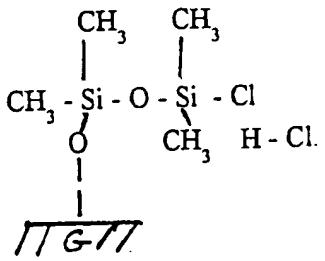


This structure then reacts with a DMS molecule as follows:

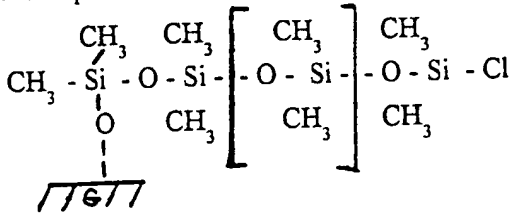
(e)



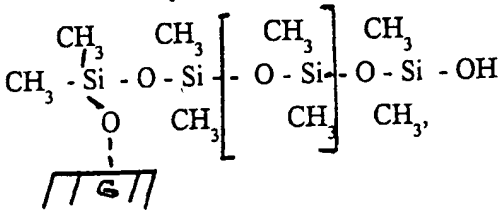
resulting in the molecule shown below:

(f) 

The process of steps (c) - (f) above is repeated about 100 times until no more water remains for steps (c) and (d). The product has the formula:

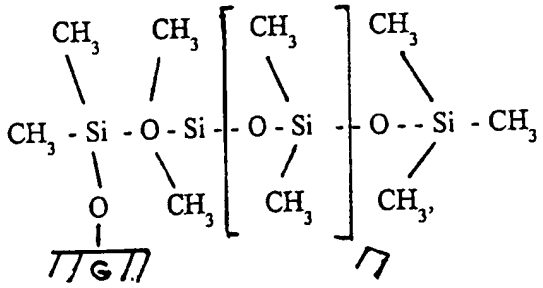
(g) 

where n is around 100 or more. The groups in the brackets are highly water repellent. However, the chlorine atom at the end of the chain slowly reacts over several hours with water vapor in the air to result in a product having the formula:

(h) 

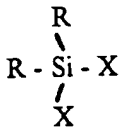
which is equivalent to the structure shown in FIG. 1. The Si - O - H group at the end of this final product is water-attracting, thus reducing the overall water repellence of the entire film, and creating a site for undesirable chemical reactions.

In the improved process of the instant invention, a surface coated with the above film is then treated with trimethylchlorosilane, which reacts with the OH group at the end of the DMS chain to produce trimethylchlorosiloxane (TMS). The final product is a film having the formula:

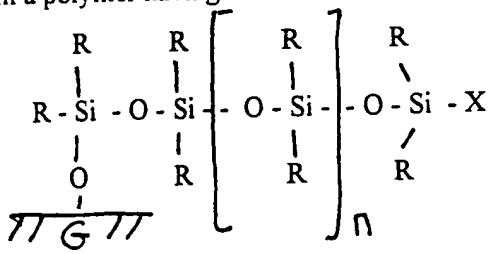
(i) 

which is equivalent to the structure shown in FIG. 2. Because the TMS group at the end of the chain is chemically inert, the water-resistance of the film is much greater than that of the prior art film.

The silicone film produced by the process of steps (a)-(i) above is one specific example of the invention, intended for water-repellent applications. In a more general case, the moistened surface G is first contacted with silane groups having the formula



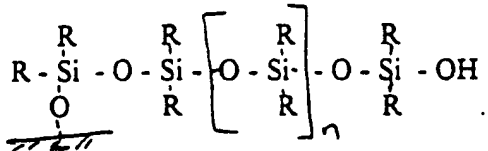
wherein R represents polar or nonpolar groups including hydrocarbons or halogenated hydrocarbons, and X is selected from the group consisting of esters, ethers, and halogens. The silane groups then react with the OH or nitrogen hydrogen bonds and water at the surface G to chemically bond the film to the surface G, in a process analogous to step (b) above. A series of reactions analogous to those shown in steps (b) - (f) above results in a polymer having the formula:



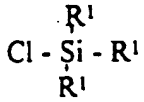
where n is around 100 or more. The X atom at the end of the chain then reacts with

water vapor

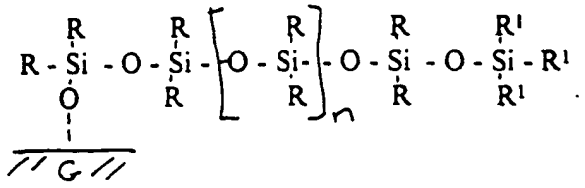
in the surrounding air resulting in a molecule having the formula:



The surface is then contacted with a capping agent having the formula:



where R^1 may include any combination of inert and reactive groups. The capping agent reacts with the OH group at the end of the chain, resulting finally in a chain having the formula:



The properties of the film manufactured using this process will depend on the choice of R and R and to a lesser extent, X. Choosing X from the chloro group gives the lowest material cost and gives a faster reaction time, while esters and ethers are less reactive but produce less troublesome coproducts and require different processing conditions.

In general, for water repellent applications, R consists of nonpolar groups and R' consists of chemically inert groups. If R consists of approximately 50% methyl groups and 50% phenyl groups, the abrasion-resistance of the film is improved. The abrasion-resistance of the film can also be improved by connecting the DMS chains with methyltrichlorosilane (which causes branched chains and additional ends). The methyltrichlorosilane would cause the chains to be tied together in a three-dimensional structure, which would resist abrasion better than a two-dimensional

structure.

For non-water repellent applications, R consists of polar groups. If R^1 is selected from chemically reactive groups, the end molecule can provide an attachment point for enzymes, chelating agents, ion exchange elements, chemical indicators and other substances.

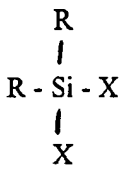
Various other modifications and variations to the embodiments herein chosen for purposes of illustration will readily occur to those skilled in the art. To the extent that such variations and modifications do not depart from the spirit of the invention, they are intended to be included within the scope thereof which is assessed only be a fair interpretation of the following claims.

Claims

1. A process for treating a surface of a substrate G containing OH or nitrogen hydrogen bonds and surrounded by air, the process including the steps of:

a) moistening the surface with water;

b) contacting the surface with silane groups having the formula

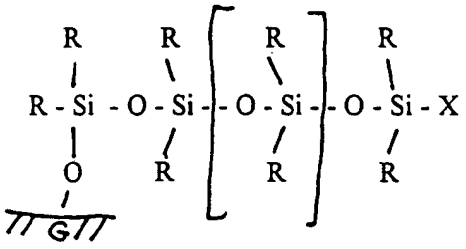


wherein

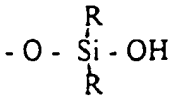
R represents polar or nonpolar groups including hydrocarbons or halogenated hydrocarbons, and

X is selected from the group consisting of esters, ethers, and halogens;

c) allowing the silane groups to react with the OH or nitrogen hydrogen bonds and water at the surface to create a film formed of chains having the formula

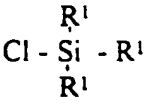


d) allowing the X atom at the end of the chain to react with water to produce a molecule having the structure



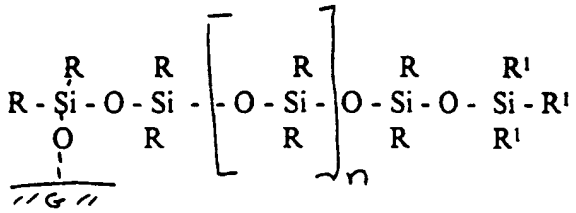
at the end of the chain; and

e) contacting the surface with a capping agent having the formula



wherein R^1 may include any combination of inert and reactive groups; and

f) allowing the capping agent to react with the molecule to result in a chain having the formula



2. The process according to Claim 1, wherein the substrate G includes silica molecules.

3. The process according to Claim 2, wherein the substrate G is formed from a material selected from the group consisting of glass, ceramics and silica-containing minerals.

4. The process according to Claim 1, wherein the substrate G includes organic molecules.

5. The process according to Claim 1, wherein R is methyl.

6. The process according to Claim 1, wherein R is selected from the group consisting of phenyl, ethyl, methyl, butyl, amyl, and larger alkyl groups.

7. The process according to Claim 1, wherein R consists of 50% methyl groups and 50% phenyl groups, resulting in improved abrasion resistance of the film.

8. The process according to Claim 1, wherein R consists of polar groups.

9. The process according to Claim 1, wherein R consists of nonpolar groups.

10. The process according to Claim 1, wherein R' consists of chemically inert groups.

11. The process according to Claim 1, wherein R' consists of chemically reactive groups.

12. The process according to Claim 1, wherein the step of contacting the surface with silane groups comprises chemically depositing the siloxane groups on the surface using a vapor machine.

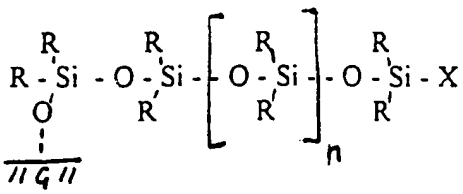
13. The process according to Claim 1, wherein the step of contacting the surface with silane groups comprises a wipe-on method.

14. The process according to Claim 1, wherein the step of contacting the surface with silane groups comprises a dipping or spraying procedure.

15. The process according to Claim 1, wherein the step of moistening the surface comprises a step of priming the surface with cyclohexylamine.

16. A process for manufacturing water-resistant glass G in an environment including air, comprising the steps of:

a) coating the glass with a somewhat water-resistant film formed of chains having the formula



wherein

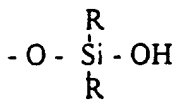
R consists of nonpolar groups,

X is selected from the groups consisting of esters, ethers and halogens, and

n is around 100 or more,

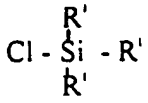
the film being chemically bonded with the glass;

b) allowing the X atom at the end of the chain to react with water to produce a molecule having the structure



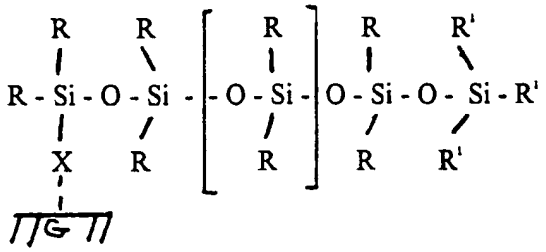
at the end of the chain; and

c) contacting the surface with a capping agent having the formula



wherein R' consists of inert groups; and

d) allowing the capping agent to react with the molecule to result in a greatly water-resistant film formed from chains having the formula



17. The process according to Claim 16, wherein the somewhat water-resistant film comprises chains of dimethylsiloxane.

18. The process according to Claim 16, wherein the capping agent is trimethylchlorosilane.

19. The process according to Claim 16, wherein R is selected from the group consisting of phenyl, ethyl, methyl, butyl, amyl and larger alkyl groups.

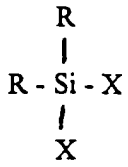
20. The method according to Claim 16, wherein R comprises approximately 50% methyl groups and 50% phenyl groups, resulting in improved abrasion-resistance of the highly water-resistant film.

21. A process for treating a surface of a substrate G containing OH or nitrogen hydrogen bonds and surrounded by air, the process including the steps of::

a) moistening the surface with water;

b) contacting the surface with silane groups having the formula

R

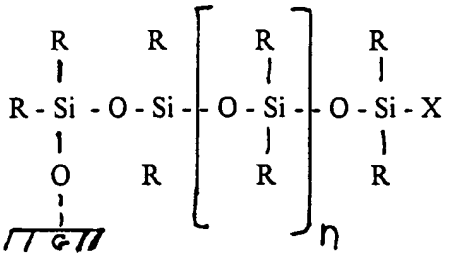


wherein

R consists of polar groups, and

X is selected from the group consisting of esters, ethers and halogens;

c) allowing the silane groups to react with the OH or nitrogen hydrogen bonds and water at the surface to create a film formed of chains having the formula

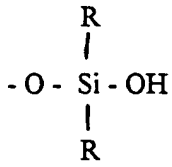


wherein

n is around 100 or more, and

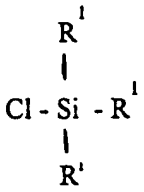
the film is chemically bonded to the surface;

d) allowing the X atom at the end of the chain to react with water in the surrounding air to produce a molecule having the structure



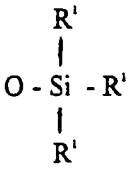
at the end of the chain; and

e) contacting the surface with a capping agent having the formula



wherein R^1 consists of chemically active groups; and

f) allowing the capping agent to react with the molecule to result in a new end molecule having the formula



wherein the new end molecule serves as a solid state ion exchanger or attachment point for chemically bound enzymes, chelating agents, dyes, chemical indicators, or the like.

22. The process according to Claim 21, wherein the substrate G includes silica molecules.

23. The process according to Claim 22, wherein the substrate G is formed from a material selected from the group consisting of glass, ceramics, and silica-containing minerals.

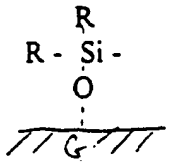
24. The process according to Claim 21, wherein the substrate G includes organic molecules.

25. The process according to Claim 21, wherein R is methyl.

26. The process according to Claim 21, wherein the film consists of chains of dimethylsiloxane.

27. A film for altering the properties of a surface G containing OH or nitrogen hydrogen bonds and surrounded by air, the film consisting of a polymer including:

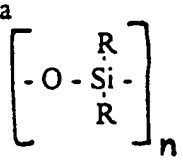
a) an anchor group having the formula



wherein

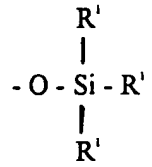
R represents polar or nonpolar groups including hydrocarbons or halogenated hydrocarbons;

b) a chain of siloxane groups having a first end and a second end, the first end of the chain being chemically bound to the silicon molecule of the anchor group, the chain having the formula



where n is around 100 or more; and

c) a terminal group of molecules chemically bound to the second end of the chain, the terminal group having the structure



where R' may include any combination of inert and reactive groups.

28. The film according to Claim 27, wherein R' is the same as R.

29. The film according to Claim 27, wherein R is methyl.

30. The film according to Claim 27, wherein R is selected from the group consisting of phenyl, ethyl, methyl, butyl, amyl, and larger alkyl groups.

31. The film according to Claim 27, wherein R consists of polar groups.

32. The film according to Claim 27, wherein R consists of nonpolar groups.

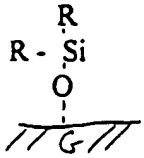
33. The film according to Claim 27, wherein R' consists of chemically inert groups.

34. The film according to Claim 27, wherein R' consists of chemically reactive groups.

35. In a process of manufacturing a water-resistant film for protecting a surface G containing OH or nitrogen hydrogen bonds wherein the process comprises

the step of coating the glass with a polymer having

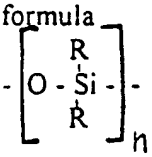
i) an anchor group having the formula



wherein

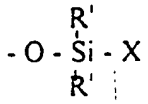
R consists of nonpolar groups, and

ii) a chain of siloxane groups, the chain having a first end and a second end, the first end of the chain being chemically bound to the silicon molecule of the anchor group, the chain having the formula



where n is around 100 or more, and

iii) a terminal group of molecules chemically bound to the second end of the chain, the terminal group having the formula



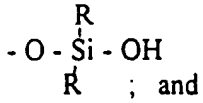
wherein

R' consists of inert groups, and

X is selected from the group consisting of esters, ethers and halogens,

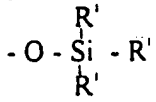
the improvement comprising the steps of

a) allowing the X atom of the terminal group to react with water to produce a new terminal group having the formula



; and

b) replacing the new terminal group with a final terminal group having the structure



wherein R' consists of inert groups.

36. The improvement according to Claim 35, wherein the siloxane groups consist of dimethylsiloxane groups.

37. The improvement according to Claim 36, wherein the final terminal group consists of a trimethylsiloxane group.

38. The improvement according to Claim 35, wherein R^1 is the same as R.

39. The improvement according to Claim 35, wherein R is selected from the group consisting of phenyl, ethyl, methyl, butyl, amyl, and larger alkyl groups.

40. The improvement according to Claim 35, wherein the step of replacing the new terminal group comprises the substeps of:

a) contacting the surface with a capping agent having the formula

$$Cl - \underset{\dot{R}^1}{\overset{R^1}{\overset{|}{\underset{|}{Si}}}} - R^1 \quad ; \text{and}$$

b) allowing the capping agent to react with the new terminal group to result in an inert final terminal group.

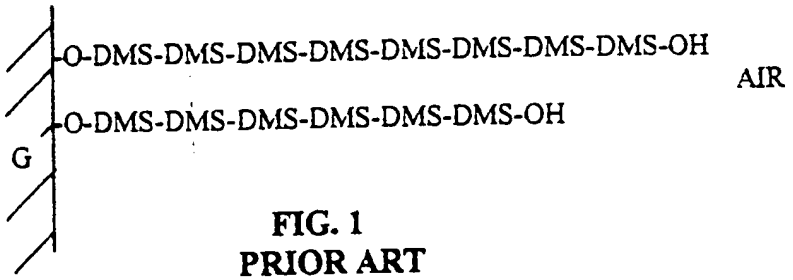


FIG. 1
PRIOR ART

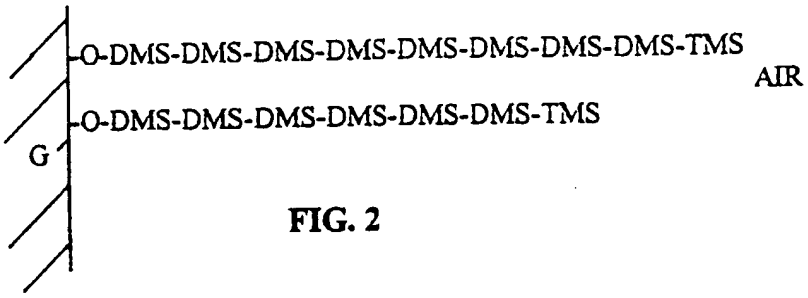


FIG. 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/01939

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B05D 1/36, 3/10; B32B 17/06, 17/10

US CL :427/387, 389.7, 402, 407.1, 407.2; 428/333, 429, 447, 451

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 427/387, 389.7, 407.2; 428/333, 429, 447, 451

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 5,451,459 (OGAWA ET AL) 19 September 1995, column 4, line 33 to column 5, line 18 and Figure 6(b).	27-34
A	US, A, 4,539,061 (SAGIV) 03 September 1985.	1-40
A	US, A, 4,263,350 (VALIMONT) 21 April 1981.	1-40
A	US, A, 5,415,927 (HIRAYAMA ET AL) 16 May 1995.	1-40

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

30 APRIL 1999

Date of mailing of the international search report

25 MAY 1999

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

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Form PCT/ISA/210 (second sheet)(July 1992)*

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/01939

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

Please See Extra Sheet.

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/01939

BOX II. OBSERVATIONS WHERE UNITY OF INVENTION WAS LACKING
This ISA found multiple inventions as follows:

I. Claims 1-15 and 21-34, drawn to a process and a film, classified in class 427, subclass 387 and in class 428, subclass 428.

II. Claims 16-20, drawn to a process classified in class 427, subclass 407.2.

III. Claims 35-40, drawn to a process classified in class 427, subclass 389.7.

The listed inventions lack unity of invention under PCT Rules 13.1 to 13.3 because there is no special technical feature which links the diverse processes of Groups I, II and III. The group I is directed to a process wherein claimed polysiloxane film is formed using silane. The group II directed to a process of forming claimed polysiloxane film on a glass surface using polysiloxane polymer. The group III is directed to a process wherein claimed anchor groups are formed on glass surface then one end of polysiloxane chains are attached to the anchor group and other end attached to a reactive terminal group finally attached terminal groups are replaced with new terminal groups.

Bob Rowan - Re: Legal Advice re Agreements

From: Bob Rowan
To: GroupIP@Foseco.com
Subject: Re: Legal Advice re Agreements
CC: Len Mitchard; Sherri Scharmach

Caroline--

I have to leave town for West Coast in a few hoours and will not be back for remainder of week. I have however, asked yet another of our partners, Sherri Scharamch, to immediately attend to this. I will also take your inquiry with me and be available for discussion at cell # 703-587-0979 (in Pacific Time zone--6 hrs diff from you, 3 from Sherri)

>>> Len Mitchard 3/26/2004 11:15:35 AM >>>
Caroline:

I have asked my partner Bob Rowan to respond to you on this. Thanks and regards.

Len

Leonard C. Mitchard
NIXON & VANDERHYE P.C.
1100 North Glebe Road, 8th Floor
Arlington, VA 22201
Tel (receptionist): +1 703-816-4000
Tel (direct): +1 703-816-4005
Fax: +1 703-816-4100
E-mail: lcm@nixonvan.com
Web site: www.nixon-vanderhye.com

>>> <GroupIP@Foseco.com> 3/25/2004 3:56:37 AM >>>

Len

I wonder if you have yet had an opportunity to review my note of 08 March please?

Many thanks in advance.

Regards

Caroline
----- Forwarded by GroupIP/FosecoInternational/Foseco on 25/03/2004 08:55

GroupIP
To: lcm@nixonvan.com@internet
08/03/2004 10:10 cc:
Subject: Legal Advice re Agreements

Len

I am not sure whether you are able to answer the following question, (it is not patent related) but John Smith has requested that I use N&V as a first point. Foseco International Limited (as you are aware!) benefits from a licence agreement from CCPI Inc. The royalty provisions of such agreement are based on a rather complicated formula which includes a "Material Cost of Sales" definition. Foseco and CCPI have recently disagreed on the calculation of such "Material Cost of Sales" and Foseco are now seeking a US legal opinion as to the interpretation of such definition. Or at least confirmation that the definition is ambiguous or open to interpretation!

The definition is as follows :

""Material Cost of Sales" shall mean the raw material and packaging costs incurred by the Licensee and its sub-licensees respectively in the manufacture of the Products, including a loss factor in respect of such raw materials of 5%. "

Given that Foseco enjoys "an exclusive licence under the Patents to manufacture, have manufactured, use and sell the Products ..." Foseco has been interpreting the definition as follows :

1. Where Foseco manufactures the products themselves, the actual cost of raw materials and packaging has been used; and
2. where Foseco has the Products manufactured by a third party (i.e. under a toll manufacture arrangement) Foseco has utilised the "bought-in" cost of the Products from such third party. Foseco does not have access to the third party's raw material costs.

CCPI insist that the second interpretation is invalid and that Foseco must ascertain the actual raw material costs of such third party manufactured products.

Are you able to advise how, under US law the definition would be interpreted? Are Foseco (or CCPI) being unreasonable?

Many thanks in advance, I look forward to hearing from you.

Best regards

Caroline O'Flynn

*****